$S = 1.256$
1295 reflections
217 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00191(F_o)^2]$
Extinction correction: Larson (1967)
Extinction coefficient: 0.012 (2)
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	1.0614 (4)	0.4048 (2)	−0.0659 (1)	0.050 (1)
C(2)	1.0640 (4)	0.4626 (2)	−0.1247 (1)	0.052 (1)
C(3)	0.8671 (3)	0.4995 (2)	−0.1423 (1)	0.041 (1)
C(4)	0.7758 (3)	0.5823 (2)	−0.0994 (1)	0.040 (1)
C(5)	0.7853 (3)	0.5291 (2)	−0.0381 (1)	0.036 (1)
C(6)	0.7056 (4)	0.6047 (2)	0.0095 (1)	0.046 (1)
C(7)	0.7008 (4)	0.5458 (2)	0.0662 (1)	0.038 (1)
C(8)	0.8007 (3)	0.4532 (2)	0.0780 (1)	0.049 (1)
C(9)	0.9412 (3)	0.4036 (2)	0.0359 (1)	0.039 (1)
C(10)	0.9797 (3)	0.4791 (2)	−0.0178 (1)	0.036 (1)
C(11)	1.1255 (4)	0.3681 (3)	0.0671 (1)	0.052 (1)
C(12)	1.0897 (4)	0.2992 (2)	0.1211 (1)	0.052 (1)
C(13)	0.9590 (4)	0.3569 (2)	0.1650 (1)	0.039 (1)
C(14)	0.7727 (4)	0.3884 (2)	0.1335 (1)	0.041 (1)
C(15)	1.0445 (4)	0.4677 (2)	0.1887 (1)	0.039 (1)
C(16)	1.2524 (4)	0.4689 (2)	0.2057 (1)	0.053 (1)
C(17)	0.9031 (4)	0.2737 (2)	0.2125 (1)	0.050 (1)
C(18)	0.5642 (4)	0.5955 (3)	−0.1157 (1)	0.063 (1)
C(19)	0.8701 (5)	0.6993 (2)	−0.1052 (1)	0.057 (1)
C(20)	1.1221 (4)	0.5730 (2)	−0.0022 (1)	0.055 (1)
O(1)	0.8805 (3)	0.5465 (2)	−0.1992 (1)	0.053 (1)
O(2)	0.9276 (3)	0.5053 (1)	0.2351 (1)	0.056 (1)
O(3)	1.2931 (4)	0.3898 (2)	0.2496 (1)	0.087 (1)
O(4)	1.0588 (4)	0.2225 (2)	0.2424 (1)	0.073 (1)

Table 2. *Selected geometric parameters (Å, °)*

C(1)—C(2)	1.530 (3)	C(2)—C(3)	1.502 (3)
C(3)—C(4)	1.539 (3)	C(4)—C(5)	1.557 (3)
C(5)—C(6)	1.530 (3)	C(6)—C(7)	1.491 (3)
C(7)—C(8)	1.332 (3)	C(8)—C(9)	1.507 (3)
C(1)—C(10)	1.533 (3)	C(5)—C(10)	1.557 (3)
C(9)—C(10)	1.559 (3)	C(9)—C(11)	1.538 (4)
C(11)—C(12)	1.518 (4)	C(12)—C(13)	1.530 (3)
C(8)—C(14)	1.513 (3)	C(13)—C(14)	1.541 (3)
C(13)—C(15)	1.548 (3)	C(15)—C(16)	1.508 (4)
C(13)—C(17)	1.532 (3)	C(4)—C(18)	1.536 (4)
C(4)—C(19)	1.545 (4)	C(10)—C(20)	1.539 (4)
C(2)—C(1)—C(10)	113.2 (2)	C(1)—C(2)—C(3)	111.3 (2)
C(2)—C(3)—C(4)	113.1 (2)	C(2)—C(3)—O(1)	107.7 (2)
C(4)—C(3)—O(1)	111.9 (2)	C(3)—C(4)—C(5)	108.3 (2)
C(3)—C(4)—C(18)	107.8 (2)	C(5)—C(4)—C(18)	107.9 (2)
C(3)—C(4)—C(19)	110.0 (2)	C(5)—C(4)—C(19)	115.4 (2)
C(18)—C(4)—C(19)	107.3 (2)	C(4)—C(5)—C(6)	113.9 (2)
C(4)—C(5)—C(10)	117.9 (2)	C(6)—C(5)—C(10)	108.9 (2)
C(5)—C(6)—C(7)	111.7 (2)	C(6)—C(7)—C(8)	123.8 (2)
C(7)—C(8)—C(9)	122.2 (2)	C(7)—C(8)—C(14)	121.8 (2)
C(9)—C(8)—C(14)	115.9 (2)	C(8)—C(9)—C(10)	113.9 (2)
C(8)—C(9)—C(11)	110.4 (2)	C(10)—C(9)—C(11)	113.0 (2)
C(1)—C(10)—C(5)	109.0 (2)	C(1)—C(10)—C(9)	108.2 (2)
C(5)—C(10)—C(9)	108.1 (2)	C(1)—C(10)—C(20)	110.3 (2)
C(5)—C(10)—C(20)	111.1 (2)	C(9)—C(10)—C(20)	110.0 (2)
C(9)—C(11)—C(12)	113.5 (2)	C(11)—C(12)—C(13)	113.9 (2)
C(12)—C(13)—C(14)	107.4 (2)	C(12)—C(13)—C(15)	112.7 (2)
C(14)—C(13)—C(15)	106.8 (2)	C(12)—C(13)—C(17)	110.0 (2)
C(14)—C(13)—C(17)	106.4 (2)	C(15)—C(13)—C(17)	113.1 (2)
C(8)—C(14)—C(13)	114.7 (2)	C(13)—C(15)—C(16)	118.3 (2)
C(13)—C(15)—O(2)	108.3 (2)	C(16)—C(15)—O(2)	110.7 (2)
C(15)—C(16)—O(3)	112.1 (2)	C(13)—C(17)—O(4)	115.4 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	H···A	D···A	D—H···A
O(4)—H···O(3)	2.583 (4)	1.68 (3)	164 (2)
O(1)—H···O(2^{i})	2.711 (3)	1.77 (3)	172 (3)
O(3)—H···O(1^{ii})	2.683 (3)	1.86 (3)	161 (3)
O(2)—H···O(4^{iii})	2.636 (3)	1.82 (2)	171 (3)

Symmetry codes: (i) $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$; (ii) $\frac{5}{2} - x, 1 - y, \frac{1}{2} + z$; (iii) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The H atoms in the CH and CH_2 groups were allowed to ride on the bonded C atom and refined.

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*, *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acetonide-3-trinervinone†

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Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, 2,2,4b′,8′,8′-pentamethyl-6,10a′-epoxyspiro[1,3-dioxepane-5,2′-perhydrophenanthren]-7′-one, $C_{23}H_{36}O_4$, which consists of three

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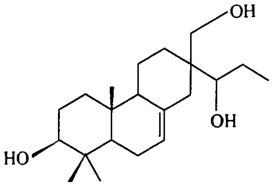
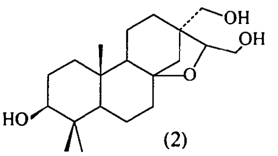
six-membered rings (*A*, *B* and *C*), and a five- and a seven-membered ring (*D* and *E*, respectively). The *C* ring is fused at C(8)—C(14)—C(13) to the *D* ring. The *A*/*B*, *B*/*C* and *D*/*E* rings are *trans*, *trans* and *cis* fused, respectively. The *A*, *B* and *C* rings adopt a distorted chair $^1C_4$ conformation. The *D* and *E* rings have envelope and half-chair $^5H_4$ conformations, respectively. The crystal structure is stabilized by van der Waals interactions. There are two C—H···O hydrogen bonds of less than 3.5 Å.

Comment

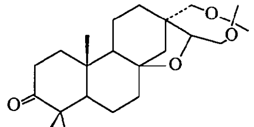
Given our interest in the phytochemical study of the Compositae family of plants found in Mexico, we have investigated the constituents of *Piqueria trinervia*, *Cav* (Jiménez & González de la Parra, 1983). A tetraol tricyclic diterpenoid, trinervinol, (1), was isolated from the flowers and leaves of this plant. During structure elucidation of (1) using chemical transformations and spectral data, compounds (2) and (3) were obtained. Trinervinol is a new diterpene with an isopimarane skeleton with a hydroxyl group at C(17). Details of the chemical transformations and spectral data of (2) and (3) are given by Jiménez & González de la Parra (1983). In this paper, the molecular structure of (3) is reported.



(1)



(2)



(3)

The Cremer & Pople (1975) ring-puckering parameters for the *A*, *B*, *C*, *D* and *E* rings are: *A*, q^2 = 0.116 (5), q^3 = 0.499 (5), Q = 0.512 (5) Å, θ = 13.1 (5), φ = −2 (2)°; *B*, q^2 = 0.162 (4), q^3 = 0.534 (4), Q = 0.558 (4) Å, θ = 16.9 (4), φ = −1 (1)°; *C*, q^2 = 0.221 (4), q^3 = 0.621 (5), Q = 0.659 (4) Å, θ = 19.5 (4), φ = −50.2 (9)°; *D*, q^2 = 0.445 (3) Å, φ^2 = 45.2 (4)°; *E*, q^2 = 0.632 (3), q^3 = 0.575 (4), Q = 0.854 (3) Å, θ = 47.7 (2), φ^2 = −35.2 (4), φ^3 = 167.2 (3)°. A similar conformation has been observed in (3*R*,5*R*,8*S*,10*R*,13*R*)-*ent*-pimara-9(11),15-dien-3-yl *p*-bromobenzoate (Soriano-García, Guerrero & Toscano, 1986) and closely resembles 11*β*-hydroxy-7-ketosandaracopimar-8(14),15-diene (Rao, Krishna & Suseela, 1985) for the C atoms in rings *A* and *B*. The molecules in the crystal are packed at normal van der Waals distances. There are two C—H···O interactions of less than 3.5 Å: C(20)···O(2) 3.187 (5) and C(23)···O(1)(x − 1, y − 1, z) 3.463 (6) Å.

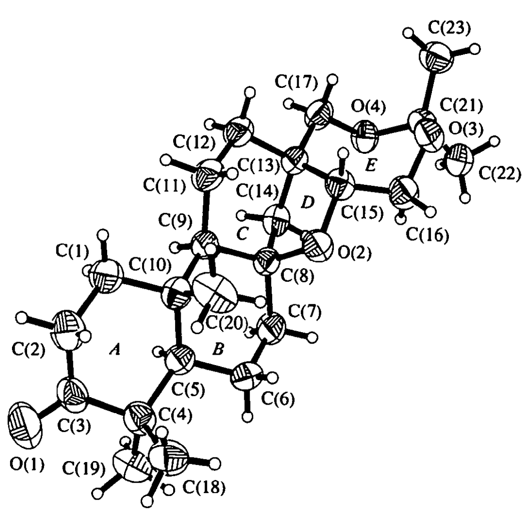


Fig. 1. The molecular structure of the title compound with the atom labelling; 50% probability displacement ellipsoids are shown.

Experimental

Compound (3) was obtained during the elucidation of (1), which was isolated from the flowers and leaves of *Piqueria trinervia*, *Cav*.

Crystal data

$C_{23}H_{36}O_4$
M_r = 376.54
Orthorhombic
$P2_12_12_1$
a = 8.664 (2) Å
b = 12.314 (4) Å
c = 19.763 (4) Å
V = 2108 (1) Å^3
Z = 4
D_x = 1.19 Mg m^{-3}
D_m = 1.20 Mg m^{-3}
D_m measured by pycnometry

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 5.0–20.2°
μ = 0.074 mm^{-1}
T = 296 K
Prism
0.60 × 0.52 × 0.30 mm
Colourless

Data collection

Nicolet *P*3/*F* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2297 measured reflections
2153 independent reflections
1742 observed reflections [$I > 2.8\sigma(I)$]
$\theta_{\max}$ = 25°

h = 0 → 10
k = 0 → 14
l = 0 → 23
2 standard reflections monitored every 150 reflections
frequency: 90 min
intensity decay: 5%

Refinement

Refinement on F
R = 0.052
wR = 0.073

$(\Delta/\sigma)_{\max}$ = 0.097
$\Delta\rho_{\max}$ = 0.223 e Å^{-3}
$\Delta\rho_{\min}$ = −0.217 e Å^{-3}

$S = 1.208$
1742 reflections
244 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00223(F_o)^2]$

Extinction correction: Larson (1967)
Extinction coefficient: 0.00062 (3)
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	1.0563 (4)	0.1689 (4)	0.8590 (3)	0.064 (2)
C(2)	1.1222 (5)	0.2827 (3)	0.8749 (3)	0.071 (2)
C(3)	1.0307 (5)	0.3702 (3)	0.8424 (2)	0.051 (1)
C(4)	0.8553 (5)	0.3693 (3)	0.8533 (2)	0.049 (1)
C(5)	0.7914 (4)	0.2499 (3)	0.8482 (2)	0.042 (1)
C(6)	0.6219 (5)	0.2388 (3)	0.8671 (2)	0.053 (1)
C(7)	0.5574 (4)	0.1333 (3)	0.8389 (2)	0.052 (1)
C(8)	0.6468 (4)	0.0314 (3)	0.8574 (2)	0.040 (1)
C(9)	0.8245 (4)	0.0478 (3)	0.8534 (2)	0.039 (1)
C(10)	0.8884 (4)	0.1570 (3)	0.8818 (2)	0.043 (1)
C(11)	0.9036 (4)	−0.0532 (3)	0.8841 (2)	0.054 (1)
C(12)	0.8316 (4)	−0.1613 (3)	0.8595 (3)	0.052 (1)
C(13)	0.6537 (4)	−0.1597 (3)	0.8592 (2)	0.041 (1)
C(14)	0.6023 (4)	−0.0657 (3)	0.8145 (2)	0.044 (1)
C(15)	0.5932 (4)	−0.1193 (3)	0.9278 (2)	0.044 (1)
C(16)	0.4265 (5)	−0.1489 (3)	0.9429 (2)	0.051 (1)
C(17)	0.5982 (4)	−0.2717 (3)	0.8388 (2)	0.049 (1)
C(18)	0.8282 (6)	0.4260 (3)	0.9217 (2)	0.064 (2)
C(19)	0.7785 (6)	0.4383 (3)	0.7979 (2)	0.063 (2)
C(20)	0.8788 (6)	0.1594 (3)	0.9605 (2)	0.065 (2)
C(21)	0.3541 (4)	−0.3178 (3)	0.8901 (2)	0.045 (1)
C(22)	0.1856 (5)	−0.2966 (4)	0.8750 (3)	0.060 (2)
C(23)	0.3819 (5)	−0.4389 (3)	0.9026 (2)	0.061 (2)
O(1)	1.0927 (4)	0.4387 (3)	0.8087 (2)	0.080 (1)
O(2)	0.6030 (3)	−0.0023 (2)	0.9253 (1)	0.047 (1)
O(3)	0.4066 (3)	−0.2641 (2)	0.9489 (1)	0.051 (1)
O(4)	0.4346 (3)	−0.2790 (2)	0.8318 (1)	0.047 (1)

Table 2. *Geometric parameters (Å, °)*

C(1)—C(2)	1.546 (6)	C(2)—C(3)	1.484 (6)
C(3)—C(4)	1.535 (6)	C(4)—C(5)	1.574 (5)
C(5)—C(6)	1.522 (5)	C(6)—C(7)	1.521 (6)
C(7)—C(8)	1.520 (5)	C(8)—C(9)	1.554 (5)
C(1)—C(10)	1.530 (5)	C(5)—C(10)	1.568 (5)
C(9)—C(10)	1.559 (5)	C(9)—C(11)	1.545 (5)
C(11)—C(12)	1.548 (5)	C(12)—C(13)	1.541 (5)
C(8)—C(14)	1.515 (5)	C(13)—C(14)	1.522 (5)
C(13)—C(15)	1.536 (5)	C(15)—C(16)	1.519 (6)
C(13)—C(17)	1.515 (5)	C(4)—C(18)	1.540 (6)
C(4)—C(19)	1.538 (6)	C(10)—C(20)	1.558 (5)
C(21)—C(22)	1.512 (5)	C(21)—C(23)	1.531 (5)
C(3)—O(1)	1.202 (5)	C(8)—O(2)	1.456 (4)
C(15)—O(2)	1.443 (4)	C(16)—O(3)	1.434 (4)
C(21)—O(3)	1.413 (5)	C(17)—O(4)	1.426 (4)
C(21)—O(4)	1.429 (5)		
C(2)—C(1)—C(10)	112.2 (3)	C(6)—C(7)—C(8)	115.5 (3)
C(2)—C(3)—C(4)	117.6 (3)	C(7)—C(8)—C(14)	112.8 (3)
C(4)—C(3)—O(1)	121.7 (4)	C(7)—C(8)—O(2)	108.9 (3)
C(3)—C(4)—C(18)	105.7 (3)	C(14)—C(8)—O(2)	103.0 (3)
C(3)—C(4)—C(19)	109.0 (3)	C(8)—C(9)—C(11)	108.3 (3)
C(18)—C(4)—C(19)	108.0 (3)	C(1)—C(10)—C(5)	108.3 (3)
C(4)—C(5)—C(10)	117.7 (3)	C(5)—C(10)—C(9)	106.6 (3)
C(5)—C(6)—C(7)	109.9 (3)	C(5)—C(10)—C(20)	112.4 (3)
C(7)—C(8)—C(9)	112.6 (3)	C(9)—C(11)—C(12)	113.0 (3)
C(9)—C(8)—C(14)	109.1 (3)	C(12)—C(13)—C(14)	107.7 (3)
C(9)—C(8)—O(2)	110.1 (3)	C(14)—C(13)—C(15)	99.5 (3)
C(8)—C(9)—C(10)	116.5 (3)	C(14)—C(13)—C(17)	116.5 (3)
C(10)—C(9)—C(11)	113.3 (3)	C(8)—C(14)—C(13)	101.6 (3)
C(1)—C(10)—C(9)	108.3 (3)	C(13)—C(15)—C(16)	114.8 (3)
C(1)—C(10)—C(20)	110.1 (3)	C(16)—C(15)—O(2)	107.6 (3)
C(9)—C(10)—C(20)	111.0 (3)	C(13)—C(17)—O(4)	113.5 (3)
C(11)—C(12)—C(13)	113.2 (3)	C(22)—C(21)—O(3)	113.1 (3)
C(12)—C(13)—C(15)	109.9 (3)	C(22)—C(21)—O(4)	104.8 (3)
C(12)—C(13)—C(17)	107.9 (3)	O(3)—C(21)—O(4)	110.5 (3)
C(15)—C(13)—C(17)	114.9 (3)	C(16)—O(3)—C(21)	115.7 (3)
C(1)—C(2)—C(3)	111.9 (4)	C(13)—C(15)—O(2)	105.9 (3)
C(2)—C(3)—O(1)	120.7 (4)	C(15)—C(16)—O(3)	111.6 (3)
C(3)—C(4)—C(5)	110.2 (3)	C(22)—C(21)—C(23)	110.6 (3)
C(5)—C(4)—C(18)	115.3 (3)	C(23)—C(21)—O(3)	105.8 (3)
C(5)—C(4)—C(19)	108.6 (3)	C(23)—C(21)—O(4)	112.2 (3)
C(4)—C(5)—C(6)	114.0 (3)	C(8)—O(2)—C(15)	109.3 (2)
C(6)—C(5)—C(10)	110.3 (3)	C(17)—O(4)—C(21)	115.3 (3)

The H atoms in the CH and CH_2 groups were allowed to ride on the bonded C atom and refined.

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*, *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Phenyl-4-imidazolidinone (*Z*)-Oxime

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Abstract

The treatment of 4-nitroimidazoles with an excess of sodium borohydride in the presence of sodium methoxide yields the (*Z*)-oximes of 4-imidazolidinones. The title compound, 1-phenyl-4-imidazolidinone (*Z*)-

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